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Ceramic Matrix Composites (CMC) Life Prediction Development—2003

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Abstract

Accurate life prediction is critical to successful use of ceramic matrix composites (CMCs). The tools to accomplish this are immature and not oriented toward the behavior of carbon fiber reinforced silicon carbide (C/SiC), the primary system of interest for many reusable and single mission launch vehicle propulsion and airframe applications. This paper describes an approach and progress made to satisfy the need to develop an integrated life prediction system that addresses mechanical durability and environmental degradation of C/SiC.

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Introduction

The complex and demanding environments of advanced airframe and propulsion systems for future space transportation vehicles are illustrated in Figure 1. The application of ceramic matrix composites (CMC) to these systems may provide benefits in terms of life, performance, temperature margin, and weight savings. For implementation of ceramics reliable performance and accurate life prediction is absolutely essential. Current state-of-the art CMC life prediction methodologies embodied in NASALife[1] and similar codes are based on empirical formulations. In general, these models have to be calibrated using experimental data. A shortcoming of these approaches is that changes in fiber architecture, constituent volume ratios, or other variables make the material system completely "new". This requires that the empirical relations be recalibrated by extensive additional experimental testing. Much of this additional cost and time can be reduced if the analytical models are physics based and placed in a micromechanics framework. Once calibrated for a specific CMC system, the predictive capability of the model can then be utilized without additional calibration.

NASALife was developed under the Enabling Propulsion Materials Project of the High Speed Research Program. Development of this code and similar codes has focused on material systems that are markedly different from the carbon fiber reinforced silicon carbide (C/SiC) composite that is the focus of this study. These codes are lacking because they are not physics-based for accurate prediction of damage due to fatigue and fracture loading conditions. They also do not account for environmental degradation effects due to water vapor attack of silica scales and carbon oxidation that are expected to be major factors in the application of C/SiC to space propulsion systems. Thus, current methods, and the underlying empirical equations upon which they are based, are inadequate for predicting the reusable life of C/SiC space propulsion hardware.

The approach outlined in this paper is designed to resolve these shortcomings. Our objective is to provide physics based models for the complex interactive mechanical and environmental degradation mechanisms that control C/SiC life, to address mechanical property measurement and prediction from a statistical point of view, and to provide the results as inputs to a parallel micro-mechanics modeling task.

Approach and Status

The overall effort focuses on providing a robust life prediction methodology that will allow confident determination of the reusable life capability of C/SiC space propulsion hardware. This will be accomplished by updating NASALife to capture the damage and degradation mechanisms associated with static and cyclic thermal and mechanical loading of C/SiC components in a high temperature, high pressure, steam containing environment. Standard C/SiC (1K T–300 fibers, plain weave, pyrolytic carbon interface, SiC matrix formed by chemical vapor infiltration, and seal coat formed by chemical vapor deposition) from GE Power Systems Composites, LLC was chosen as the baseline material for this study. Enhanced C/SiC, with and without a life enhancing coating, is also being tested.

Life Model

Physics based, probabilistic lifing models are being pursued. The models will address issues inherently related to composite materials – stochastic characterization of strength, life, and orthotropic material response. Stress rupture tests are being carried out, and fatigue testing will be carried out, in appropriate environments in support of model calibration and validation. The lifing models developed will be implemented in NASALife. A parallel effort for a micro-mechanics (fiber /coating /matrix) based approach to predict stiffness, strength, and life at the coupon level is also being pursued.[2, 3, 4] These on-going tasks have led to a library of computer codes developed specifically for the design of CMCs, and they will be adapted to C/SiC to provide state of the art design tools.

Lifing schemes, such as those contained within NASALife and currently employed for CMC, are adapted from models originally developed for design with metals. These traditional models are comprised of modified Miner's rules, rain-flow calculations, empirical knockdown factors, safety factors, etc. Under this task, a probabilistic residual strength model is being pursued. Residual strength is taken as the damage metric for stress rupture and mechanical fatigue life models. Initial static strength, intermediate residual strength, and time or cycles to failure are all treated as random variables with similar distributions (see Figure 2). In addition, efforts are underway to develop physics-based models at the fiber/matrix level for life determination, and environmental effects. In the meantime, the residual strength model utilizes empirical relationships where needed, but is open to modification and incorporation of new models, such as micro-mechanical models and models for environmental degradation, as they become available. Some of the initial results will be shown in the section on Mechanical Testing.

Oxidation

Oxidation is one key aspect of the environmental attack problem. It arises because C/SiC composites have a microcracked SiC matrix in the as-produced condition. As a result, the pyrolytic carbon coating on the fibers and the carbon fibers themselves are subject to oxidation attack when the cracks are open.[5, 6, 7] The details of this fiber attack are illustrated in Figure 3. This degradation mechanism occurs at temperatures below the composite fabrication temperature under zero stress conditions, and at all elevated temperatures sufficient for oxidation of the fibers (>400 ^{o}C) when stress sufficient to open matrix cracks is applied. Figure 4 illustrates the role of thermal expansion mismatch between the SiC matrix and the C fibers, and of applied stress on crack opening. Since oxidizing conditions are expected to be present in the service environment of most C/SiC components, prediction of oxidation attack is a key ingredient of the life prediction model. A more thorough understanding of the effects of environment, temperature, and stress on the degradation of carbon fibers is being developed so that material limitations can be better identified and methods of improving oxidation resistance can be addressed. The development of a physics and experimentally based fiber oxidation model is being pursued as depicted in Figure 5. It incorporates such variables as reaction rate, diffusion coefficient, temperature, and oxygen partial pressure. It tracks the recession of an array of fibers in a cracked matrix so that the oxidation kinetics involved in carbon fiber degradation can be studied. Oxidation studies, stress rupture tests, and microscopy are being conducted to aid in the development of the model. During the past year experiments were carried out to determine the oxidation activation energies for T–300 carbon fiber and pyrolytic carbon interface materials as illustrated in Figures 6 and 7, respectively. The two regimes for T–300 oxidation agree with other results from literature in which two regimes were observed in the temperature range of 500-900 ^{o}C[8] and one was observed at temperatures from 600-950 ^{o}C.[9]

Mechanical Testing

The test plan for tensile, stress-rupture, and fatigue testing was formulated to satisfy several requirements: (1) calibration and verification of the probabilistic residual strength (PRS) model, (2) assessment of usable service life for various conditions (i.e. temperature, stress, and environment) for C/SiC, and (3) determination of the effect of alternative fiber architecture on material behavior and model capability. One study conducted under this effort examined the effect of specimen width on stress rupture life. Data are shown in Figure 8 for tests at 800 and 1200 ^{o}C. The fact that life increases dramatically with increasing specimen width is encouraging and indicates that component life will likely be significantly longer than predicted by test data generated on the narrow specimens. It also indicates that it is important to vary gage width as part of C/SiC material characterization. Finally, the gage width results illustrate the shortcomings of the seal coating process as is evident from the microstructures in Figure 9. Attack at machined and seal-coated edges and corners is much more severe than at normally processed surfaces. This is illustrated quantitatively in Figure 10 for the 1200 ^{o}C data. The relation is nonlinear. The rate of attack at edges is about three times the rate on large surfaces. However, since there is far more surface area than edge area, the highest volume of carbon fiber consumption occurs from the large flat surfaces. In a large panel edge, damage would become insignificant from a structural standpoint as illustrated in Figure 11. However, performance integrity and sealing between panels would be compromised.

The performance of standard C/SiC and enhanced C/SiC in air is shown in Figure 12 as a function of temperature. The enhanced C/SiC has a matrix that is modified with B_4C particulates to allow low temperature glass formation and crack sealing. Enhanced C/SiC outperforms the standard C/SiC at 800 and 1200 ^{o}C. Tests at other temperatures are planned.

Lives of standard C/SiC in air are too short for model development data. Thus an artificial condition (1000 ppm O_2 in argon) was selected to give a more readily measured life distribution. At this reduced oxygen partial pressure condition, calibration tests were run at 800 and 1200 ^{o}C. To verify the model a series of stress-rupture tests were run at 30 ksi. Results are plotted along with model predictions of probability of failure in

Figure 13. At the critical low probability of failure tail of the distribution, the predictions are in good agreement with the data at both temperatures. The situation with regard to residual strength prediction is not as good, as shown in Figures 14. The measured median residual strength is about 35 ksi after 15 hours of stress rupture exposure at 30 ksi whereas the model predicts a much more gradual strength reduction with the median strength not far below the as-received strength. The situation is similar after 7.5 hours. Figure 15 is a plot of residual strength versus time to failure for interrupted tests run at 30 ksi and for life tests run at the indicated stress equal to the residual strength. From this curve a number of degradation models were formulated and are in the process of being evaluated. Also, testing at 800 ^{o}C is underway to allow further development of the residual strength part of the model.

Stress-rupture tests to determine the effect of moisture on C/SiC performance were carried out in the apparatus partially depicted in Figure 16. Results at 600 and 1200 ^{o}C are presented in Figures 17 and 18, respectively. At 600 ^{o}C, 20% steam in argon is benign in comparison to air. At 1200 ^{o}C, steam is about as aggressive as air. Vacuum run out data are shown for comparison. These results can be understood from the fiber oxidation data shown in Figure 19. At 1200 ^{o}C, fiber oxidation in air and in 20% H_2O in Ar is about the same. At 600 ^{o}C, oxidation of T–300 still occurs rapidly in air, but in 20% H_2O in Ar the reaction kinetics limit the reaction rate to near zero.

<u>Water Vapor Attack</u>

The reaction of silica scales with water vapor is the most straightforward aspect of the environmental attack problem to characterize and model because stress state interactions are insignificant. Current state of the art consists of both experimental data and a model for SiC and Si_3N_4 recession due to formation of volatile silicon hydroxides in combustion conditions typical of aircraft engine.[10] The model predicts material recession rates as a function of water vapor partial pressure, total pressure, gas velocity, and material temperature. In this task, the model is being extended to pressures, gas chemistries, gas velocities, and material temperatures typical of the rocket engine environment. High pressure, low velocity tests will be run upstream of a nozzle throat at various O_2/H_2 (O/F, oxidizer/fuel) mixture ratios. Initial experiments were run under less than ideal fuel-rich combustion conditions that led to a non-uniform temperature distribution. Weight change results indicated that H_2O vapor attack is taking place. Thickness measurements at specific locations are shown in Figure 20. Water vapor attack appears to be more aggressive at the higher fuel-rich O/F and temperature location. Currently the combustor and nozzle assembly is being redesigned to achieve more uniform flame conditions.

Concluding Remarks

Life prediction for C/SiC is a complex problem involving many interactive mechanisms. The plan outlined here will analyze mechanisms in isolation as well as their interactions, develop mechanistic lifing models, and develop understanding of the importance of statistics in C/SiC behavior.

Progress has been made in all aspects of the plan:

- Steam effects: Preliminary tests indicate that we will see an effect consistent with past experience. A test set-up with a more uniform combustion profile will be on line in 2003. The kinetics of water vapor reaction with carbon fibers is slow at 600 ^{o}C, but comparable to air attack at 1200 ^{o}C.

- Oxidation model: The activation energies for T–300 and pyrolytic carbon were determined. Crack opening as a function of temperature and stress was calculated. Detailed microscopy of oxidized specimens is being carried out to develop the oxidation model.

- Mechanical property tests and life model: Initial results are very encouraging except for residual strength prediction. Edge oxidation of seal coated specimens has been investigated by testing specimens of varying gage width. The mitigating effect of steam observed in fiber oxidation studies has also been observed in stress rupture tests.

Future efforts will include architectural effects, enhanced coatings, biaxial tests, and low cycle fatigue. Modeling will need to account for combined effects.

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Propulsion

- High temperatures (1650 to 3000 °C)
  - Low and intermediate temperatures can also be a problem
- High pressures (e.g. to ~ 4.2 MPa (~ 6000psi))
- Severe chemical environments
  - Steam
  - Oxygen rich or fuel rich
  - Hydrogen
- High velocity
- Exposure cycles from minutes in rockets to ~hours in some combined cycle approaches
- Severe thermal transients and gradients

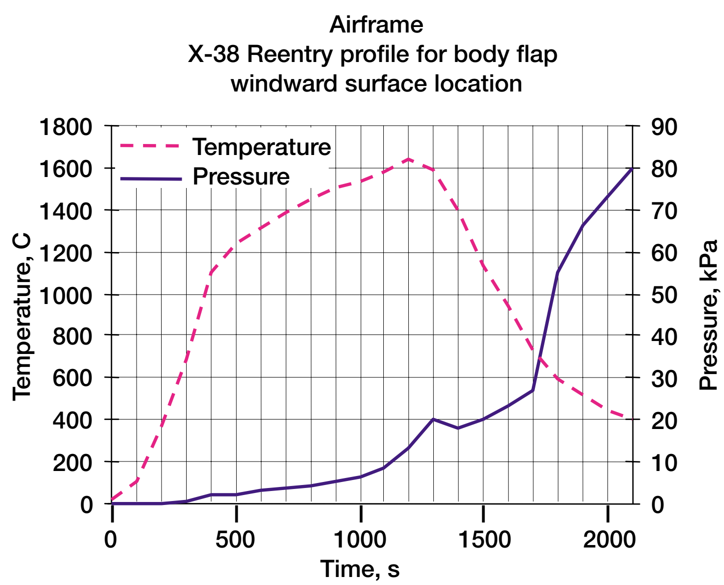


Figure 1.—Demanding environments push CMC materials limits.

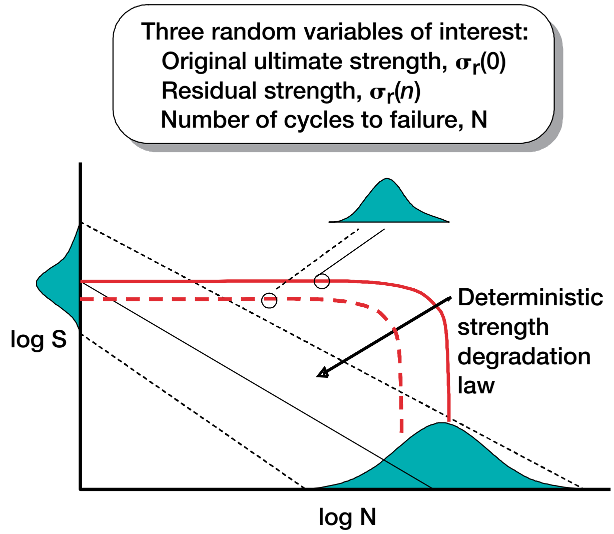


Figure 2.—Probabilistic model development for C/SiC.

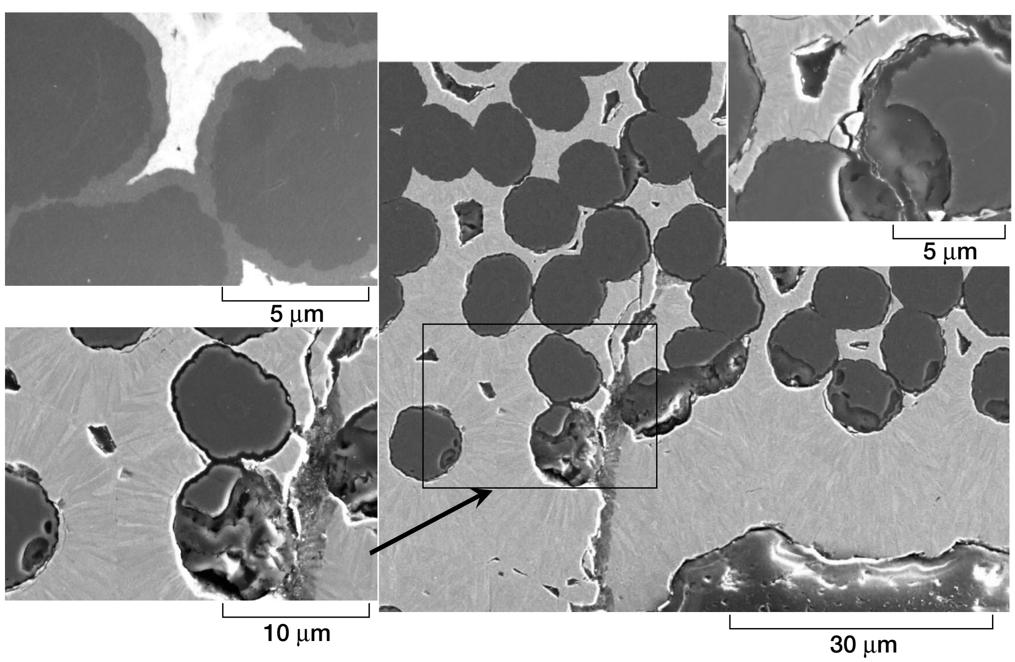


Figure 3.—Internal oxidation of C/SiC tested at 800 °C.

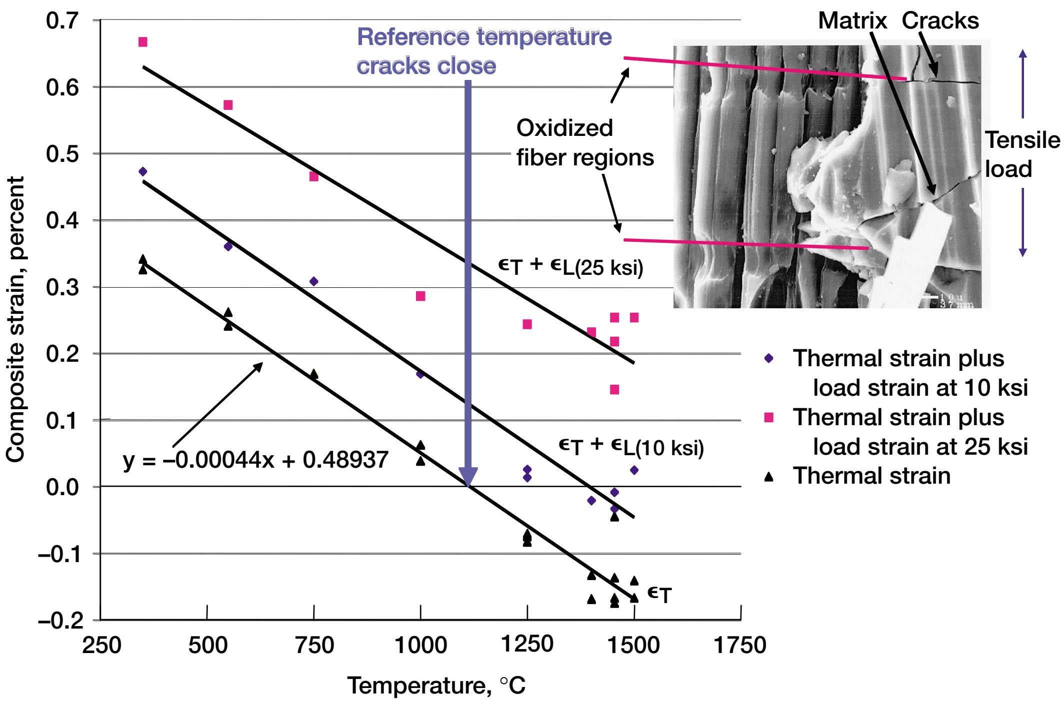


Figure 4.—Crack opening determined by load and thermal strain.

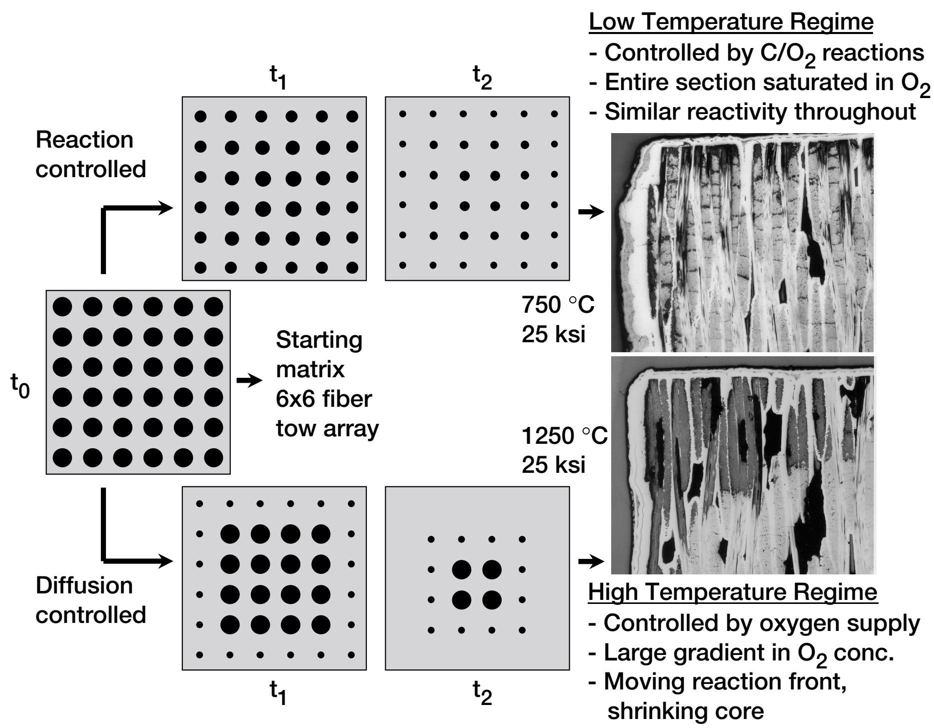


Figure 5.—Carbon fiber attack mechanism is temperature dependent.

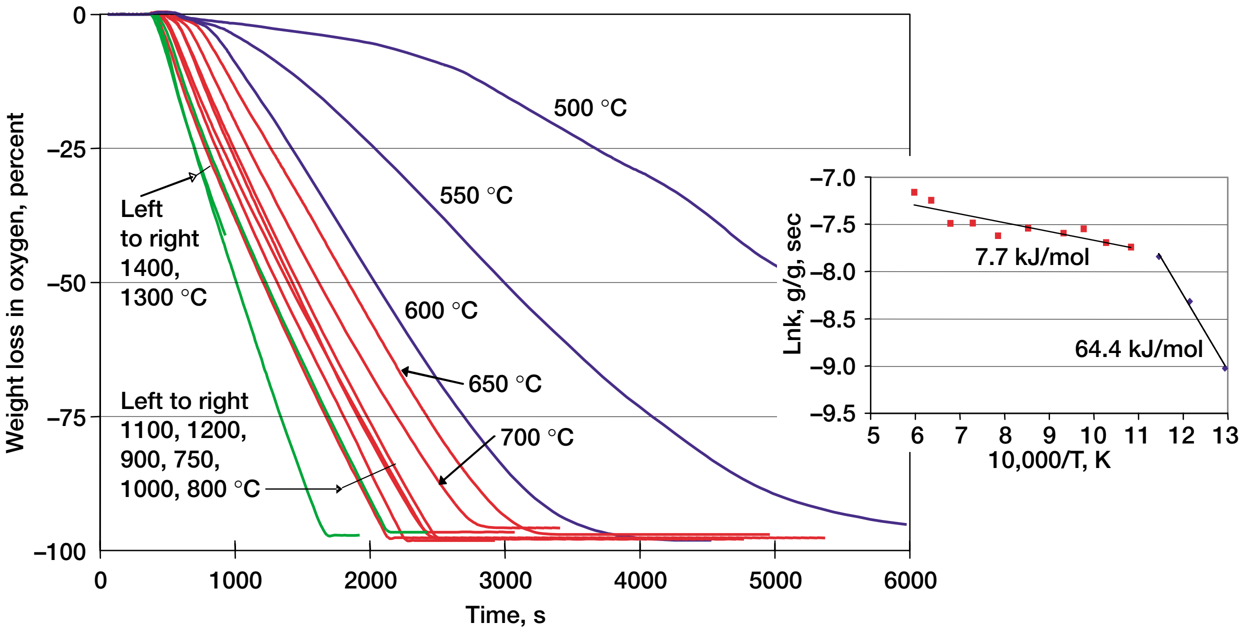


Figure 6.—TGA results for uncoated T-300 carbon fiber tow.

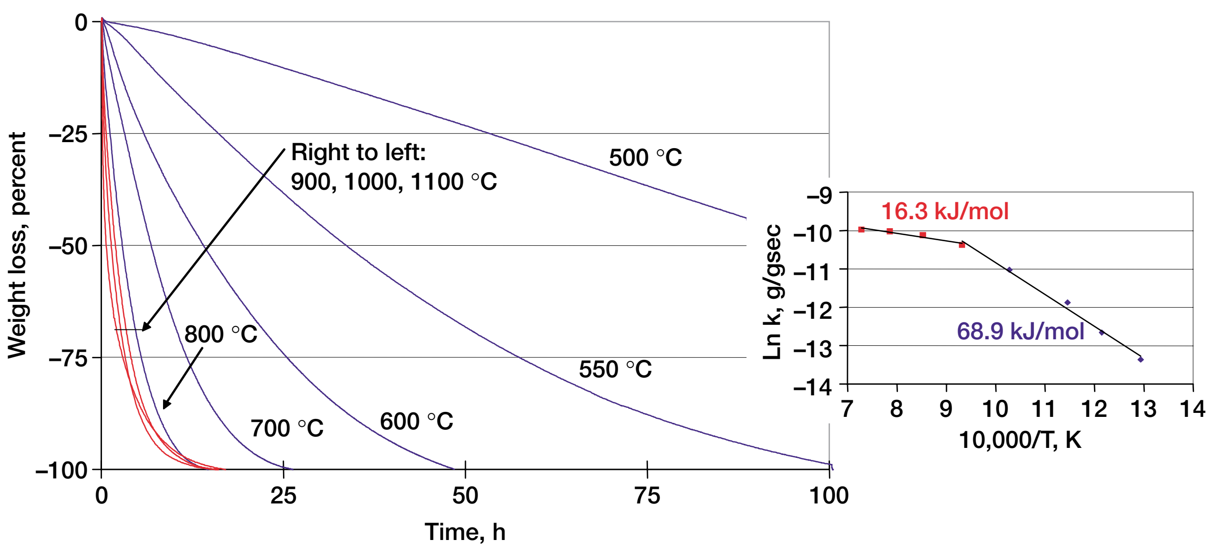
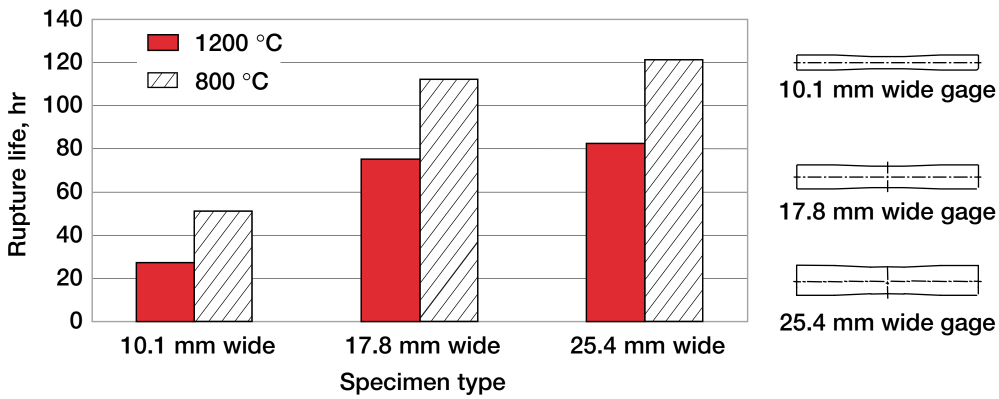
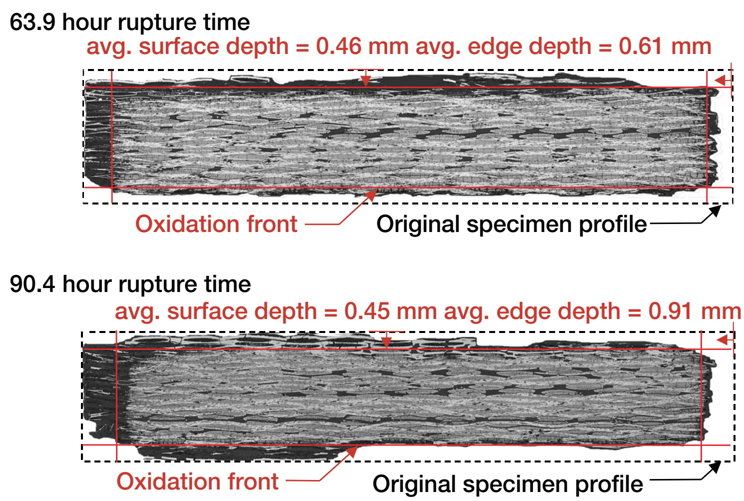


Figure 7.—Activation energy for pyro-carbon interphase oxidation. SiC/SiC coupons with machined edges: the pyro-carbon interphase is oxidized in a flowing oxygen environment. The interphase is 5 vol % of the composite (approximately 0.08 grams).



- Average life at 800 °C is about 50% longer than at 1200 °C.
- 17.8-mm wide specimens resulted in about a 2.5 x increase in life compared to 10.1-mm wide specimens.
- Data suggests that material volume and edge effects need to be incorporated in life prediction models.

Figure 8.—Effect of specimen volume on C/SiC stress-rupture life 30 ksi, 1000 ppm O_2/Ar.



17.8-mm wide specimens had less scatter in rupture life than 10.1-mm. Reduced scatter attributed to reduced influence of edge oxidation.

Figure 9.—Oxidation damage of 17.8-mm wide specimen at 1200 °C, 30 ksi, 1000 ppm O_2/Ar.

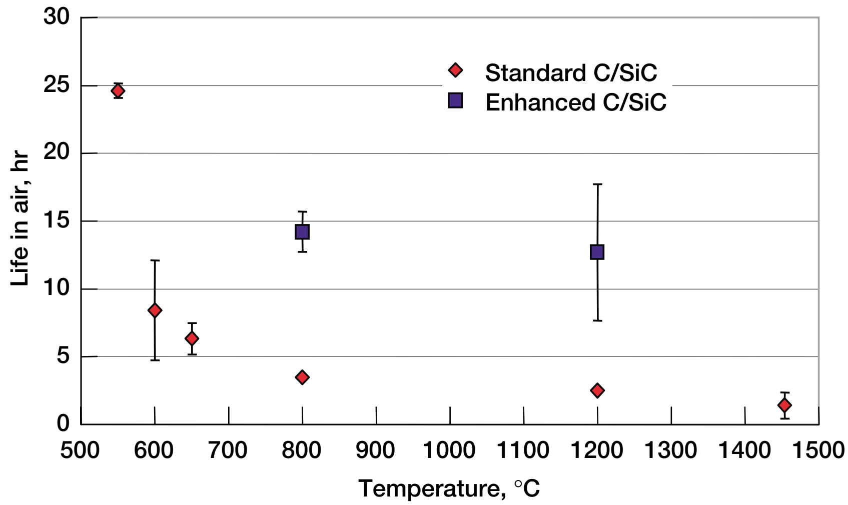


Figure 12.—Stress rupture life for C/SiC as a function of temperature (all tests conducted in air at 10 ksi stress).

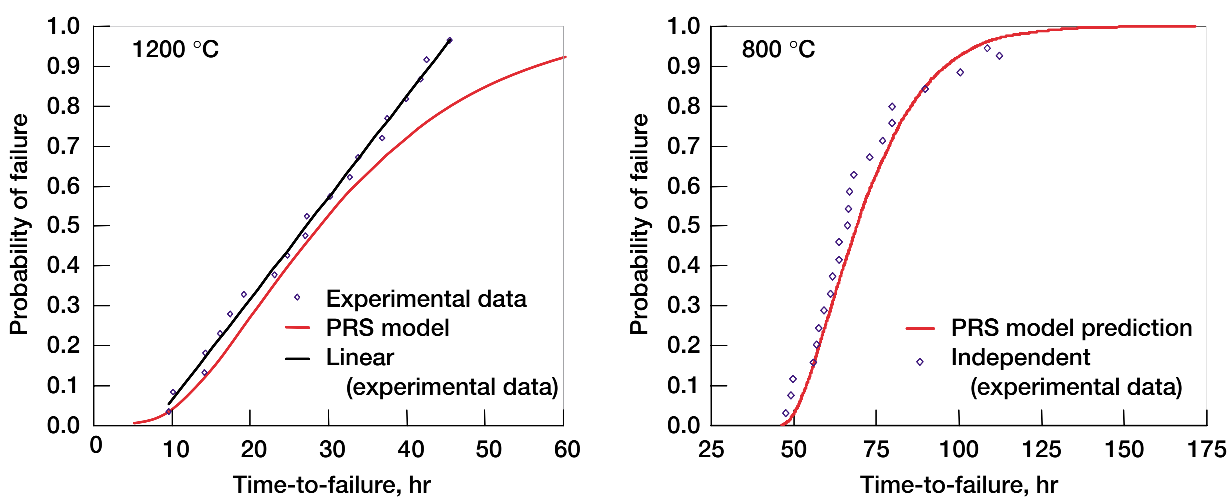


Figure 13.—Cumulative failure distribution curves for C/SiC (30 ksi, 1000 ppm O_2).

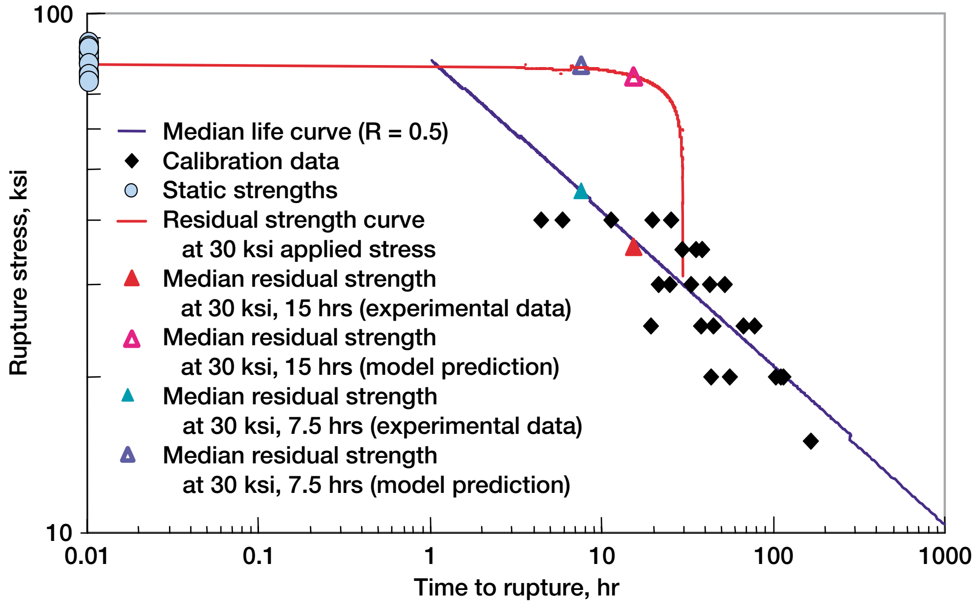


Figure 14.—Predicted residual strength for C/SiC.

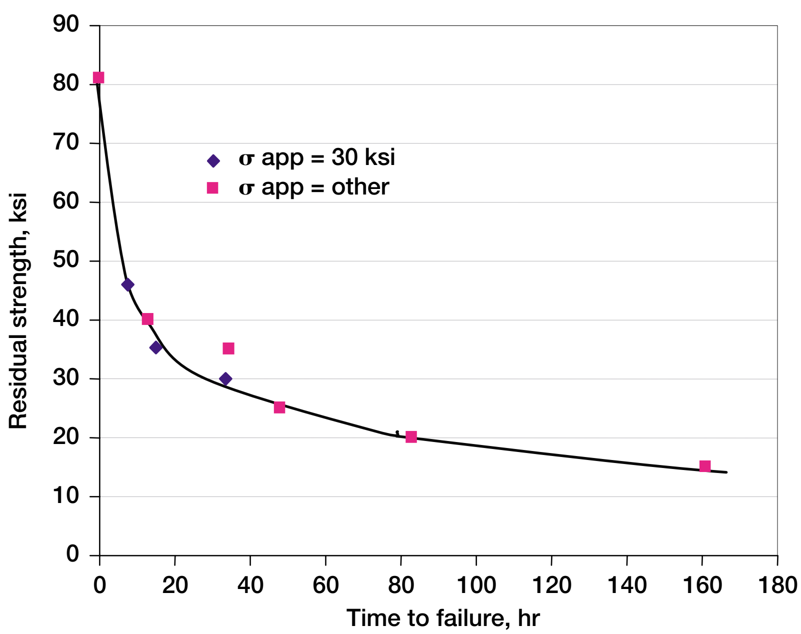


Figure 15.—Residual strength trend at 1200 °C, 1000 ppm O_2.

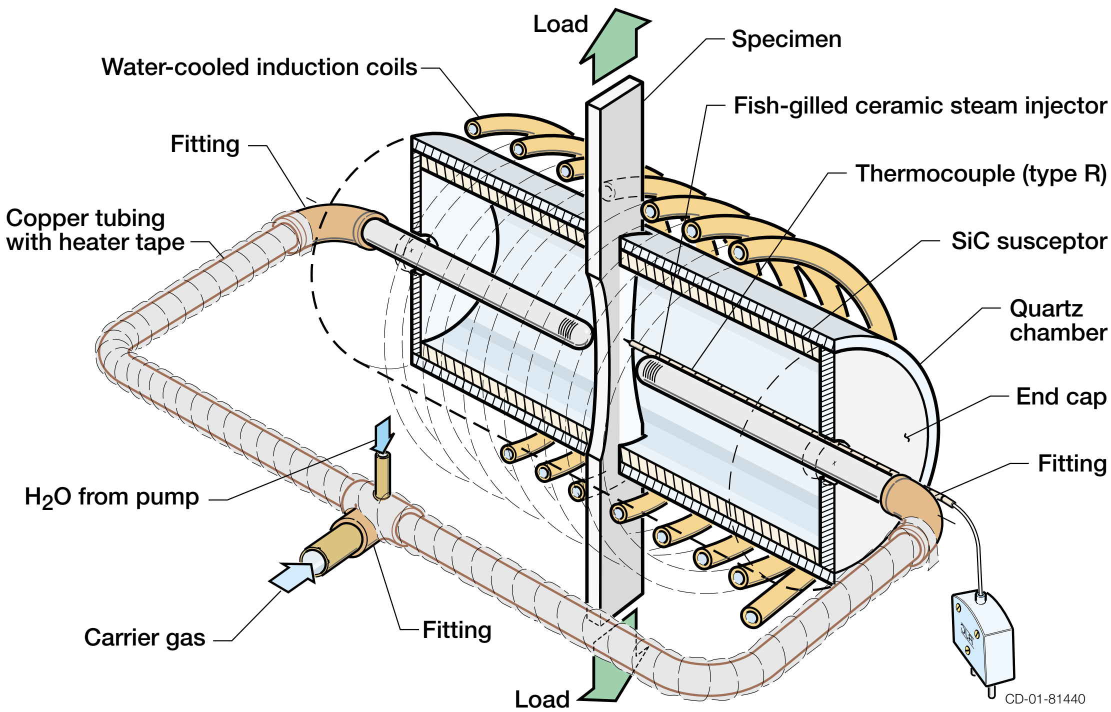
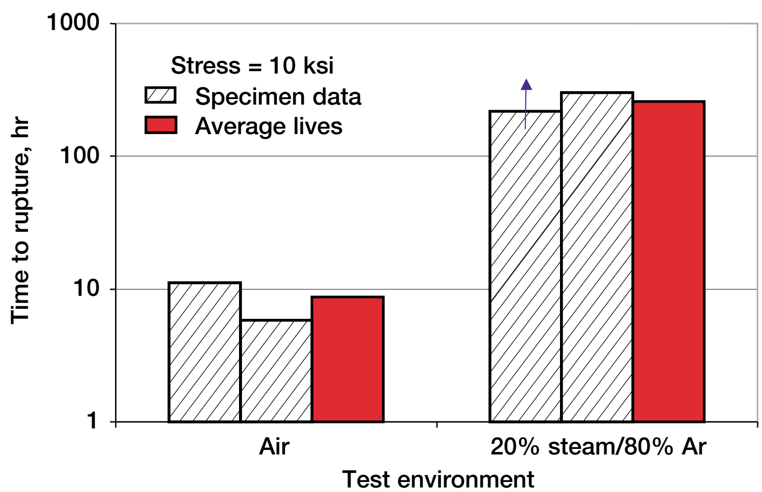
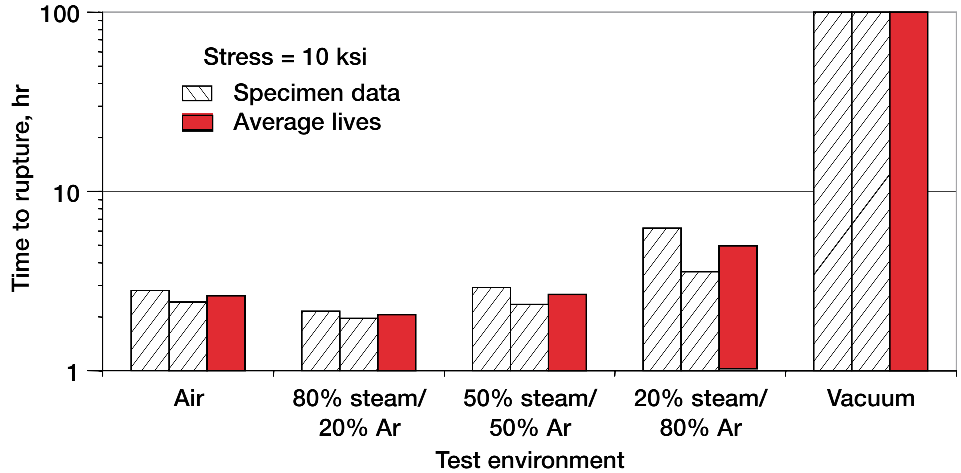


Figure 16.—Stress-rupture testing of C/SiCin steam/argon environments at ambient pressure.



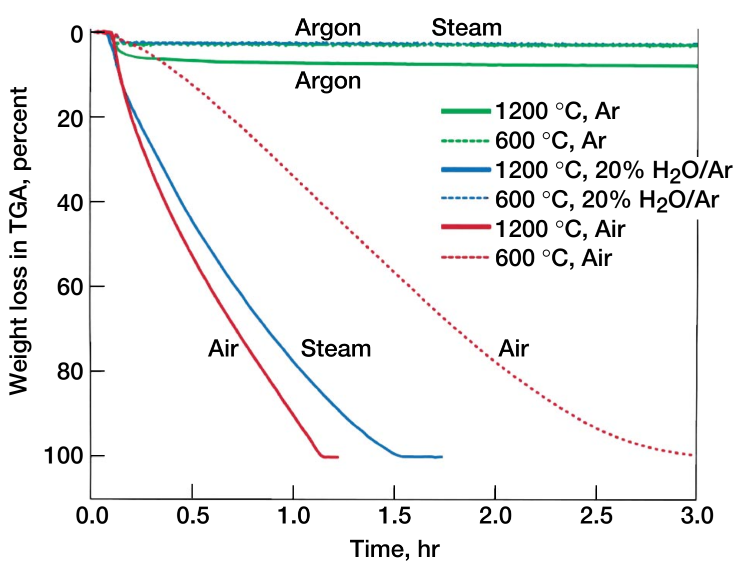
- Average life in air was 8.5 hours.
- Life in steam environment was 30 times longer than in air.

Figure 17.—Effect of environment on stress rupture life of C/SiC at 600 °C.



- Average life in 20% steam/80% Ar (5 hrs) is about twice that in other steam environments (about 2.5 hrs).
- Tests in vacuum stopped after 100 hrs, prior to specimen failure.
- 600 °C lives longer than 1200 °C, especially in steam (250 versus 5 hrs).

Figure 18.—Effect of environment on stress rupture life of C/SiC at 1200 °C.



- Weight loss in inert environment (argon) is negligible.
- At 1200 °C, carbon fiber weight loss kinetics in air and steam due to oxidation are rapid; fibers are consumed in 1.5 hours.
- At 600 °C in steam, weight loss is negligible, while in air, rapid weight loss occurs.

Figure 19.—T-300 carbon fiber oxidation kinetics.

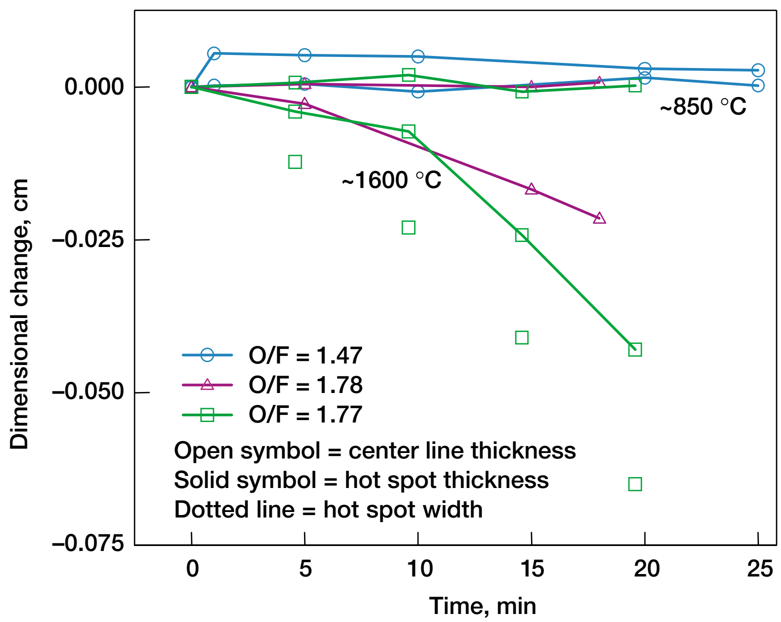


Figure 20.—Preliminary tests show recession of thick SiC seal coat in O/H combustion rig.

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13. ABSTRACT ***(Maximum 200 words)***

Accurate life prediction is critical to successful use of ceramic matrix composites (CMCs). The tools to accomplish this are immature and not oriented toward the behavior of carbon fiber reinforced silicon carbide (C/SiC), the primary system of interest for many reusable and single mission launch vehicle propulsion and airframe applications. This paper describes an approach and progress made to satisfy the need to develop an integrated life prediction system that addresses mechanical durability and environmental degradation of C/SiC.

14. SUBJECT TERMS

Ceramic matrix composites; Silicon carbide; Carbon fibers; Life prediction; Creep; Fatigue; Oxidation; Design; Prediction analysis techniques; Life (durability)

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